

Vapor Phase Lubrication for Advanced Surety Components

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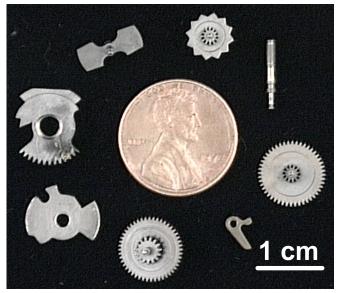
> Sandia LDRD Day 14 September 2009







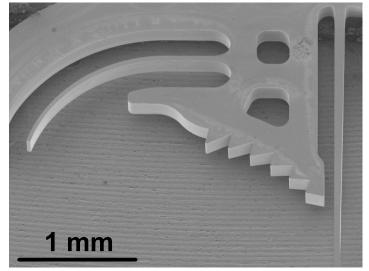
Problem - Lubrication Will Become Increasingly Challenging As Component Dimensions Shrink



MoS₂-lubricated parts from electromechanical stronglink

Lubricating small parts of complex shape is a challenge at present.

As size decreases, existing lubrication approaches are not feasible.



Future components will contain even smaller metallic parts





Problem – Robust Surface Treatment for MicroElectroMechanical Systems (MEMS) is Needed

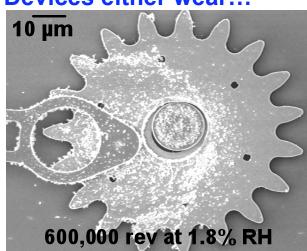
Chemisorbed monolayers for MEMS are easily damaged by contact, without sliding D.A. Hook, S.J. Timpe, M.T. Dugger and J. Krim, J. Appl. Phys., **104** (2008) p. 034303

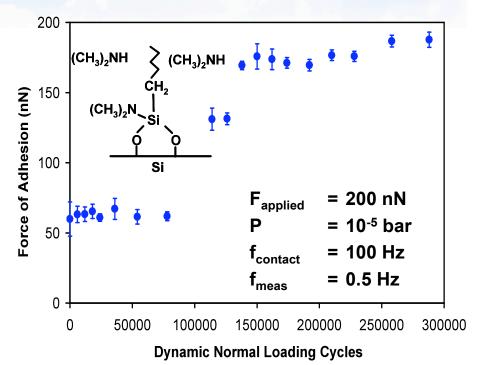
Chemisorbed monolayers are adequate release compounds, but poor lubricants.



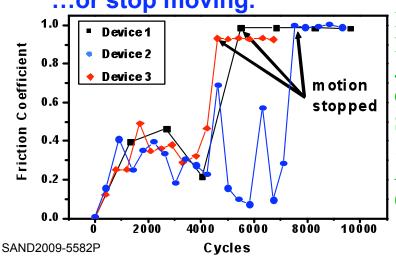
Devices either wear...

D. Tanner et. al, *Intl*. Reliability **Physics** Symposium, 1998.





...or stop moving.

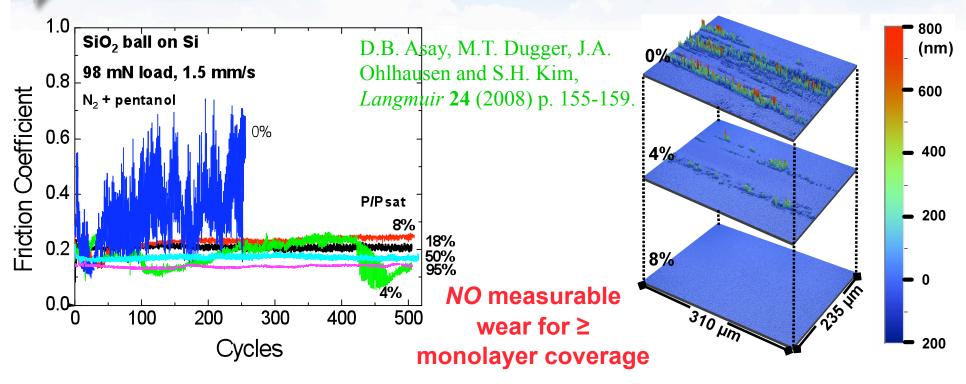


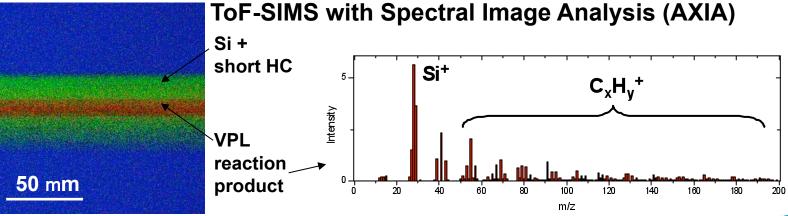
D.B. Asay, M.T. Dugger, J.A. Ohlhausen and S.H. Kim, **Tribology** Letters 29 (2008) p. 67



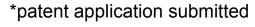


Discovery* - Vapor Phase Lubrication Reduces Friction and Wear During Sliding of Silicon











Our Approach Involves Mechanistic Understanding, Vapor Delivery, and Demonstrating Performance

Goal Develop automatically delivered, self-healing vapor lubrication system for small parts of complex shape, particularly suited to weapon surety applications

Task 1: Examine Reaction Mechanisms & Optimize Chemistry

- vary substrates and vapor to test reaction hypotheses
- determine friction response, chemistry and structure of the contact surface (ToF-SIMS, synchrotron, FTIR, etc.)
- select thermochemical properties for desired operating conditions (T, pressure) and stability with aging

Task 2: Develop Vapor Delivery System

- develop a practical vapor delivery scheme for component application
- explore A) controlled-release matrix and B) in-situ generation

Task 3: Investigate Component Performance and Aging

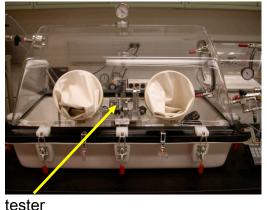
- use mechanism test beds to evaluate performance in STS environments
- determine compatibility with other materials (e.g. organic), effects on electrical contacts, and performance changes with accelerated aging



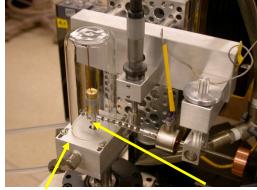


Polymerization is Not Required to Achieve Low **Wear Performance**

From ~40,000 cm³, environment around tester



To ~ 2 cm³. tester around environment



chamber lid

ball holder

ToF-SIMS with

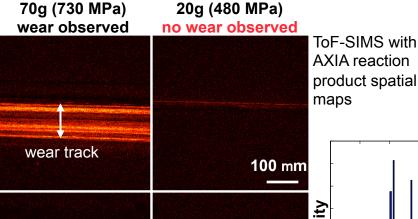
maps

- maintains control of environment
- small volume = rapid equilibration
- easy to clean; resists contamination
- amenable to more diverse vapor chemistries

Reaction product accompanies surface damage

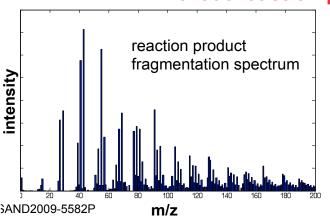
- previously, no wear for P/P_{sat} > monolayer
- found that higher contact pressure requires higher lubricant vapor pressure to avoid wear
- I wearless sliding is achievable without reaction product formation

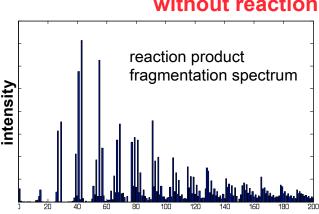
Ar+ 50% pentanol



10% pentanol









Lubrication Mechanism

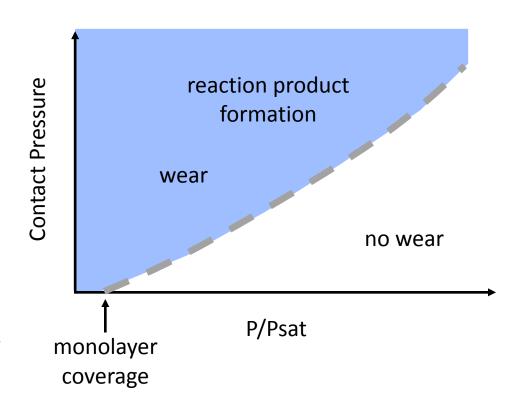
Case 1:

Low vapor concentration, high contact pressure

oligomer formation and deposition

Case 2: High vapor concentration

- no oligomer, but low friction surface is produced
- continuous supply of at least a monolayer

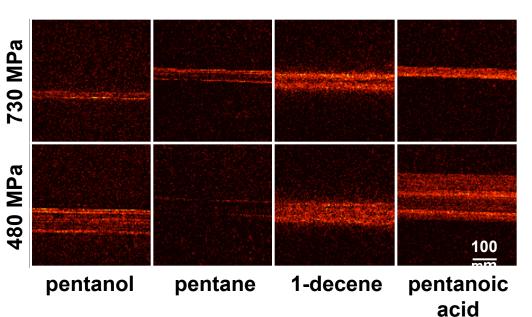


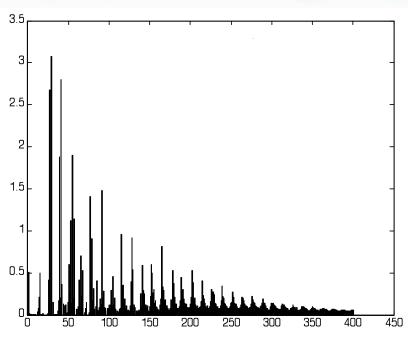
- Reaction product is not required for lubrication
 - good news for MEMS!
- This trend also seen using IR adsorption to track reaction products (collaboration with S. Kim, Penn State Chem. E. group)





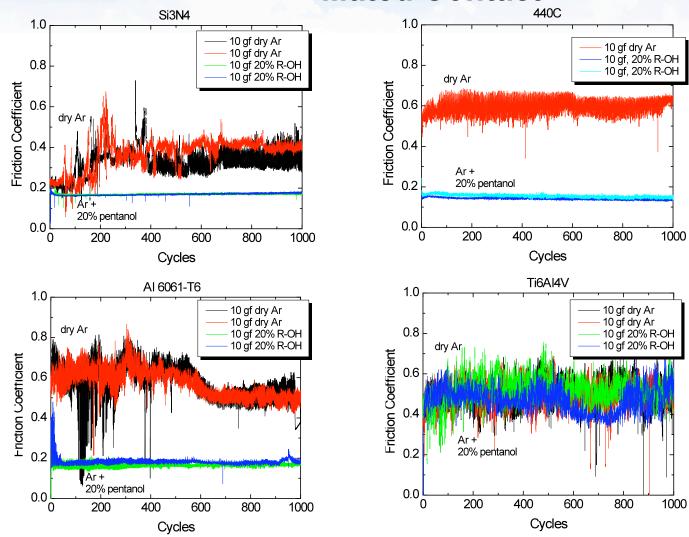
Common Polymer Fragments





- Low order fragments (<77) are CxHy, where x<=6
- High order fragments are mostly aromatic units (those <200 shown at right)

20% Psat Pentanol on Other Substrates in Self-Mated Contact



- alcohol lubricates some metal oxides as well as silicon
- no effect on titania surface





Reaction Mechanism Case 1

Several substrates and small molecules were evaluated to determine if oligomers were formed at low vapor pressure with high contact pressure

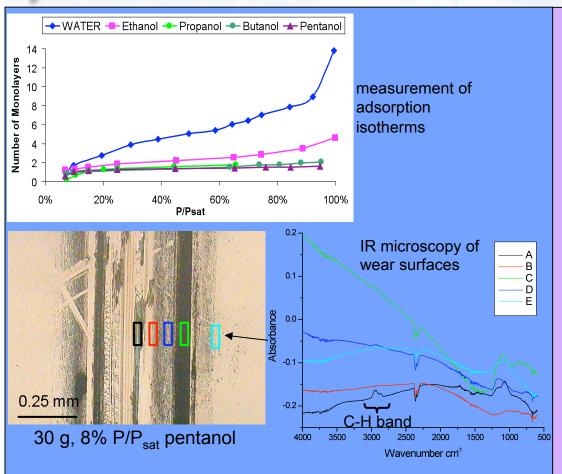
	Predicte	Predicted Oligomer formation			Observed Oligomer formation		
	Acid	Base	Free Radical	Acid	Base		
OH	Yes	Yes	Yes	Yes	Yes	Reaction Surfaces changed to	
	No	No	No*	Yes			
	Yes	Yes	Yes	Yes		create acidic or basic	
ОН	No	Yes	No	No		conditions	

^{*}normally aliphatic compounds do not react under free radical conditions





Collaborations Provide Additional Data, Hypotheses, Students, and Leverage Funding



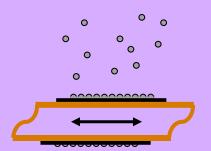
Collaboration with Prof. Seong Kim at Penn State

- NSF project on adhesion and friction in microsystems
- grad student at Sandia for summer

Collaboration with Prof. Jackie Krim at NC State

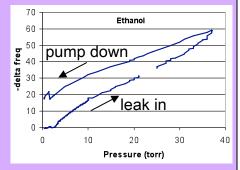
- AFOSR project on friction in extreme environments
- recent emphasis on vapor lubrication





quartz crystal microbalance studies of vapor uptake and slip on surface

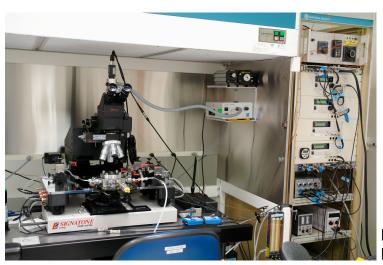
frequency hysteresis may be due to alcohol storage in monolayer-coated surface







MEMS Device Testing in Controlled Environments

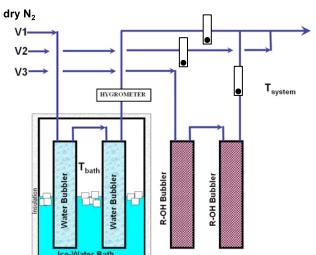


- Device packaged in 24-pin DIP
- Process image data to give forces

- adhesion
$$F_{ad} = F_{unload} - F_{load} + F_{r}$$

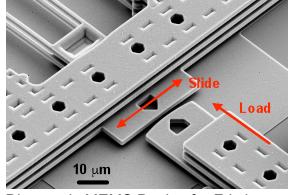
 $= a(V_c^2 - V_p^2) + kx$
- static friction $F_{fr} = F_{push} - F_{pull} - F_r$
 $= a(V_{slip}^2)$
- dynamic friction $F_d = kDx$

Probe Station and Drive Electronics





Timed Image Capture
Displacement vs Input (V)

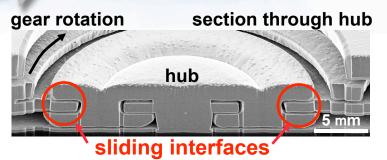


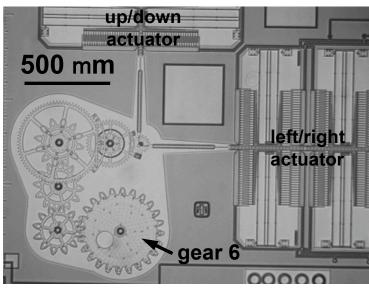
Diagnostic MEMS Device for Friction and Adhesion Measurements

MEMS Environmental Test Chamber



Operating Life of Gear Train Increased With Vapor Phase Lubrication



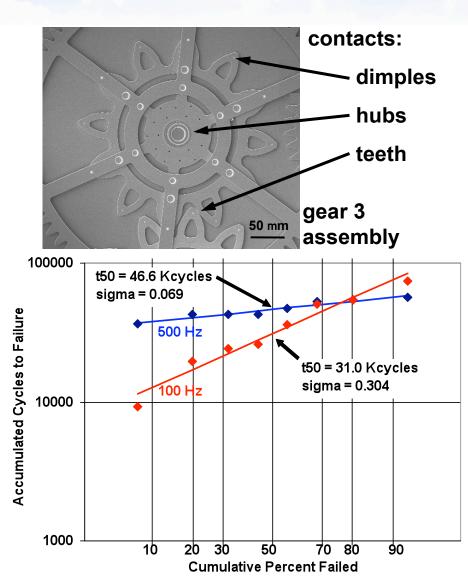


DMS prototype gear train

FOTAS monolayer alone, $t_{50} = 4.7 \times 10^4$

With VPL, device was stopped at 4.8x10⁸ cycles *without failure*

1000 ppmv pentanol



D. Tanner, Sandia

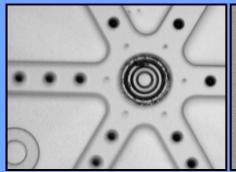




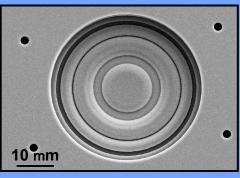


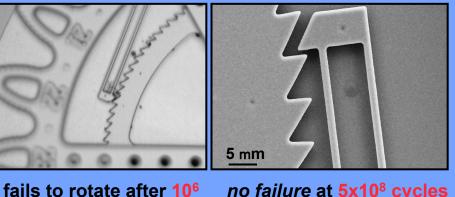
VPL is Effective on MEMS Devices with Thermal Actuators

polycrystalline silicon Rotary Actuator



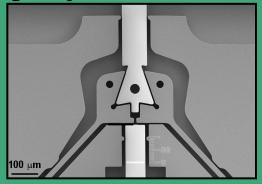
cycles in 3%RH air



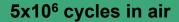














5x10⁶ cycles in 20% Psat pentanol

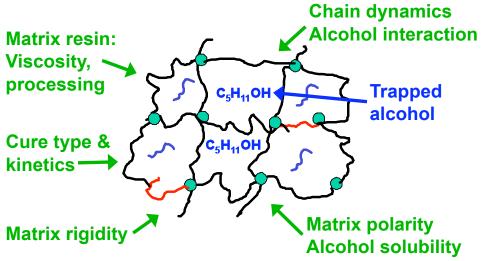
 VPL with pentanol produces extraordinary operating life in a variety of MEMS devices





Vapor Delivery Approach A: Controlled Release Matrix

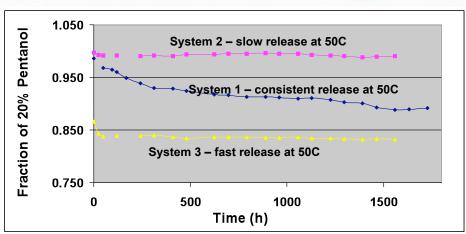
Develop a polymer matrix pre-loaded with pentanol that delivers vapors via permeation



Many design parameters are available to tailor matrix-alcohol interactions

$$y_{lv}P = x_{lp}\gamma_{lp}P_l^{sat}$$

mole fraction in polymer, x_{lp} polymer-precursor interactions, \mathbf{g}_{lp} lubricant vapor pressure, $P_l^{\,sat}$



system 1 - High Tg epoxies; rigid with low flexibility amines

system 2 - Low Tg epoxies; flexible with low flexibility amines

system 3 - sub RT Tg epoxies; flexible with acrylics and low flexibility amines

 gradual pentanol release by polymer matrices has been demonstrated

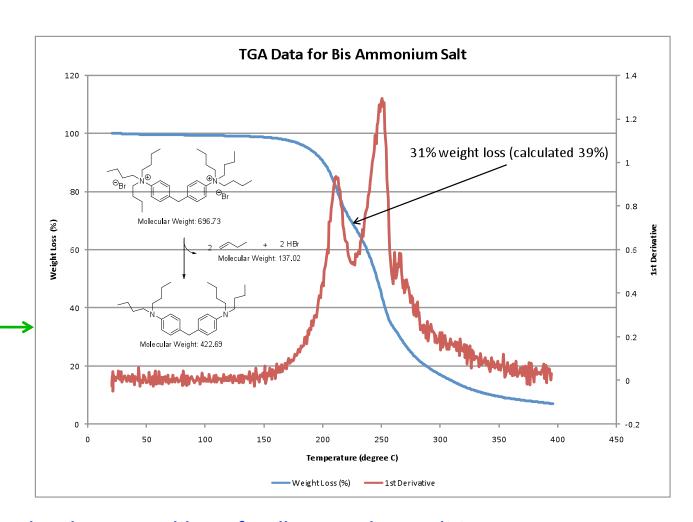




Vapor Delivery Approach B: In-Situ Generation

Explored several thermally driven reactions to deliver a vapor as needed:

- 1) Retro-ene reaction
 - Deliver alcohols or thiols
- 2) Hoffman elimination reaction
 - Deliver olefins
- 3) Thermally removable protecting group chemistry
 - Deliver olefins



TGA data shows the decomposition of a diammonium salt to produce 1-butene

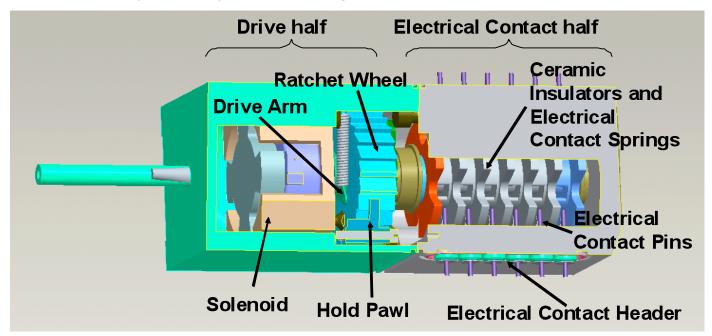




Mechanism Testing Using ECS Prototype

Electrical Contact Stronglink prototype hardware selected for VPL testing

- represents key functionality of existing surety mechanism designs
- used previously to study switch design options





- Nine units opened and inspected
 - -ratchet wheels and pawls redesigned (to be replaceable)
- Test plan developed
 - -minimum operate time; minimum operate V
 - -wear of contact surfaces
 - -electrical contact resistance
- SAND2009-5582P materials compatibility

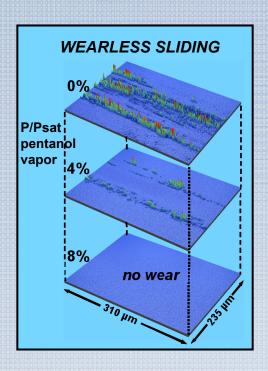


National Nuclear Security Administration

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Summary - Vapor Phase Lubrication for Advanced Surety Components



Key Accomplishments

- Extraordinary operating life demonstrated using pentanol vapor in silicon surety prototypes
- Lubrication achievable without reaction product accumulation
- Feasibility of controlled vapor delivery demonstrated

Significance of Results

- First effective lubrication scheme for microsystems is being developed
- Design barrier against sliding surfaces in silicon has been removed
- Tribology results suggest similar impact on metallic mechanisms

Vapor Phase Lubrication will potentially revolutionize surface treatment of small machines to protect against wear

Enables highly reliable, long life micro-mechanical systems for national defense and industrial applications

Sandia National Laboratories

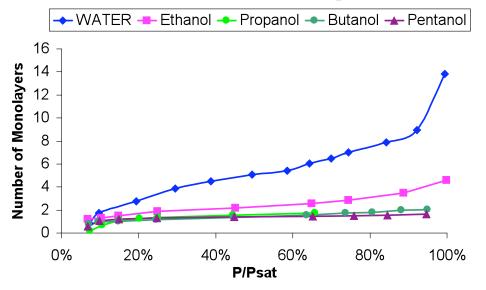
Backup Slides



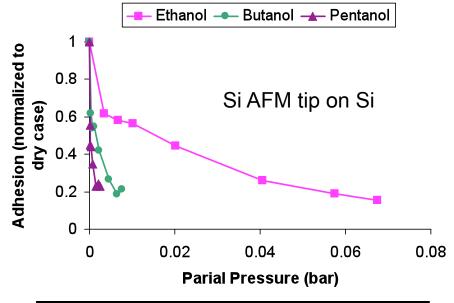


Alcohols Reduce Adhesion Between Silicon Surfaces

K. Strawhecker, D.B. Asay, J. McKinney and S.H. Kim, *Trib. Lett.* **19** (2005) p.17-21



D.B. Asay and S.H. Kim, *Langmuir* **23** (2007) pp. 12174 - 12178



Adsorbed film thickness by ATR-FTIR

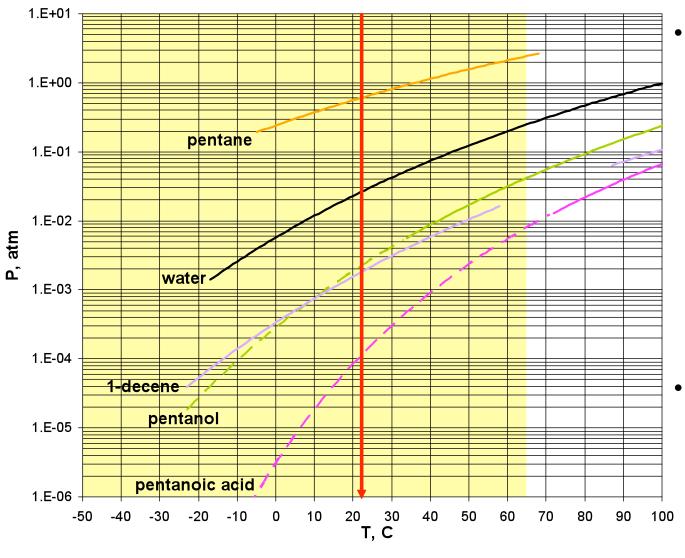
for C ≥ 2, adsorb 1-2 monolayers at 0.1
 < P/P_{sat} < 0.9

	▼ (erg/cm²)	V(cm³/mol)
water	72.8	18.1
ethanol	21.8	58.7
1-butanol	24.6	91.2
1-pentanol	24.9	108.7

 Lower surface tension, limited adsorbed film thickness, and high molar volume of alcohols limit capillary adhesion

SAND2009-5582P

The Vapor Pressure of These Liquids Varies by Orders of Magnitude



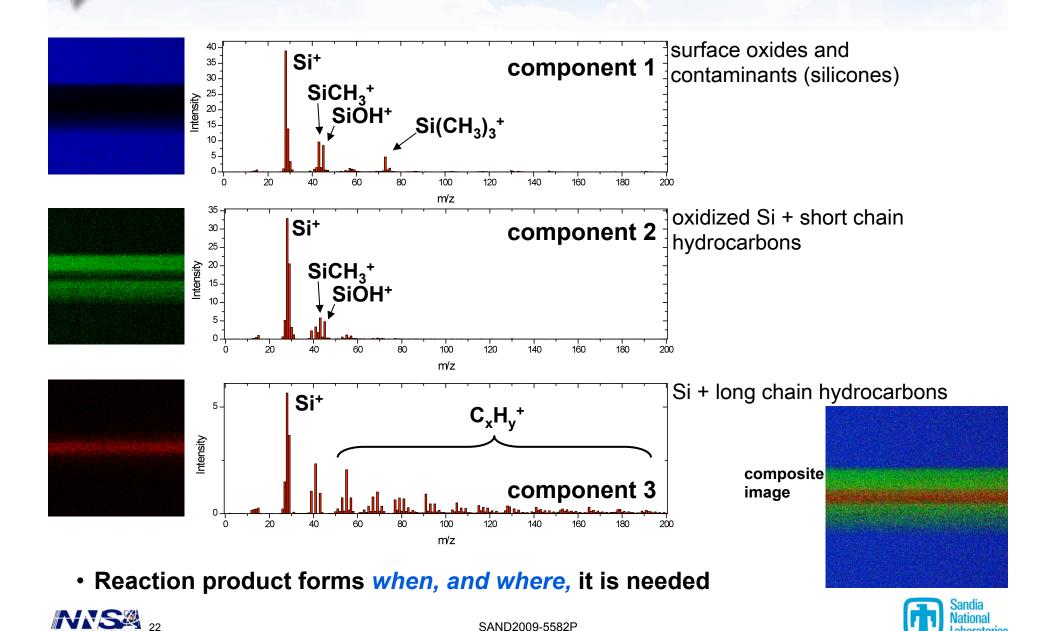
Compared to pentanol at RT:

- 1-decene has similar vapor pressure
- acid has 10x lower vapor pressure
- methyl has 100x higher vapor pressure
- Operation over wide temperature range is a challenge

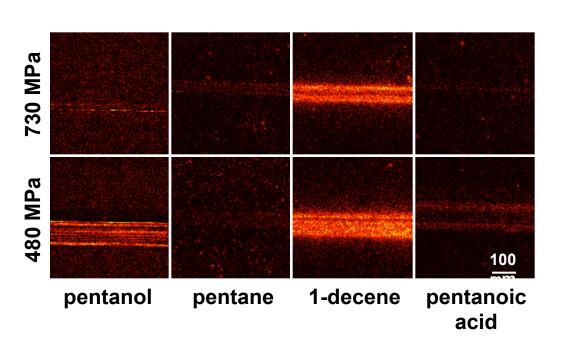


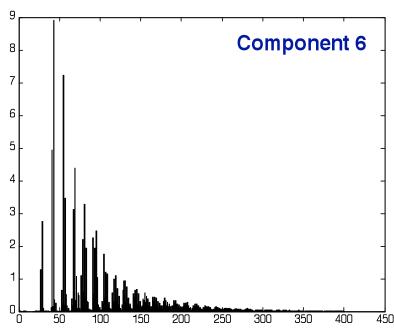


High MW Product Forms in the Contact



1-Decene Related





• CxHy fragmentation, with single and double bonds, and no aromatics





VPL is Effective on Devices with Thermal Actuators

Rotary actuator operates
 ~10⁶ cycles in 3%RH air
 cycles with VPL by pentanol at P/P_{sat}=0.2, without failure

